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(71)Applicant : NGK INSULATORS LTD

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(72)Inventor : KASAI YOSHIYUKI  
KUMAZAWA KAZUHIKO  
KOTANI WATARU

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## (54) CORDIERITE CERAMIC FILTER AND ITS PREPARATION

### (57)Abstract:

**PURPOSE:** To provide a cordierite ceramic filter with excellent regeneratability on regeneration by back washing, a high mechanical strength and lightweight and compact and a method for preparing it.

**CONSTITUTION:** A cordierite powder with a porosity of at least 30% is used as an aggregate and a raw material for making cordierite, a pore making agent, a molding auxiliary and a solvent are incorporated therein and mixing and kneading are performed and the mixture is extruded into a honeycomb shape to perform calcination after molding. The mean fine pore diameter of a filter is made within (a mean diameter of the aggregate) $\times 0.15 \pm 5\mu\text{m}$  and the porosity of the whole filter is made at least 30% and the compression strength in the flow path direction is made at least 100kgf/cm<sup>2</sup>. In addition, the thickness of a partitioning for the flow path is made 300-2,000 $\mu\text{m}$  and the porosity of the cross section of the filter part is made 63-71%.

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**CLAIMS**

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[Claim(s)]

[Claim 1] For the both ends, in the nature ceramic filter of cordierite for separate solid phase out of a fluid, this filter consist of combination of the nature SERAMMIKU aggregate of cordierite, a passage septum be form in a honeycomb configuration, the eye closure be give alternately, the average pore size of a filter be less than [ of the filter aggregate / pitch-diameter x0.15\*\*5micrometer ], the porosity of the whole filter be 30% or more, and the compressive strength of the direction of passage be 100 kgf/cm2. Nature ceramic filter of cordierite characterize by be above.

[Claim 2] The nature ceramic filter of cordierite according to claim 1 characterized by for the thickness of a passage septum being 300-2000 micrometers, and the hole density of a filter cross section being 63 - 71%.

[Claim 3] The manufacture approach of the nature ceramic filter of cordierite characterized by kneading by adding a shaping assistant and a solvent to the cordierite powder 100 weight section of 30% or more of porosity used as the aggregate, the cordierite-ized raw material 5 used as bridge formation of the aggregate - 30 weight sections, and below the ostomy agent 30 weight section, and mixing, and carrying out baking to a honeycomb configuration after extrusion molding.

[Claim 4] The manufacture approach of the nature ceramic filter of cordierite according to claim 3 characterized by the mean particle diameter of cordierite powder being 20% or less of an extrusion dice slit.

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the nature ceramic filter of cordierite which was suitable as a hot filter for gas, and its manufacture approach.

[0002]

[Description of the Prior Art] The porous honeycomb filter which gives a filtering function to fluids, such as gas, is variously proposed by making the septum of the honeycomb structure object which consists of cordierite as a porous ceramic filter pass in recent years. Since it excels in thermal resistance compared with other ingredients, the cordierite ceramics is used in comparatively high temperature and the location for which high thermal shock resistance is needed, for example, is used as three way component catalyst support of an automobile.

[0003] Since playback of a filter was performed by the playback approach by combustion within a ceramic filter, the thing for the particle uptake in the conventional nature ceramic filter of cordierite, especially the exhaust gas discharged from a diesel power plant had a problem in prolonged stable use from problems, such as partial degradation and an erosion, arising. Although the playback approach by back wash pulse air was proposed in order to solve these problems, there were various problems -- the ability to regenerate at the time of back wash playback will not be inadequate, or will not have the level which mechanical strength can carry in an automobile, or since these are covered, a system will become large.

[0004] Although the filter has been obtained by the conventional nature ceramic filter of cordierite adding a shaping assistant by using talc, a kaolin, an alumina, etc. as the main raw material, calcinating it after extrusion molding on the other hand, and performing the eye closure, in JP,61-129015,A, the technique of improving the filter engine performance by setting the number ratio of pores of the stoma of 5-40 micrometers of apertures and a 40-100-micrometer osculum to 1:5-40 is exhibited about the pore which exists in the front face of a filter septum. However, this is a technique on condition of a combustion playback system, playback by back wash pulse air is not considered, and sufficient playback is not performed even if it carries out back wash pulse regeneration of the filter indicated here as it is.

[0005] As a filter of a back wash playback type, the method of calcinating with binding material, such as a kind or two or more sorts of ceramic fines which are chosen from cordierite, beta-SUPONJUMEN, aluminum titanate, a mullite, a zirconia, silicon carbide, silicon nitride, etc. or clay, and a frit, and obtaining a ceramic filter is indicated by JP,63-31517,A. Mechanical strength sufficient by this approach was not obtained, playback by back wash pulse air was not fully made, but there was a problem that the filter in which it carries in an automobile and useful uptake time amount is shown was not obtained.

[0006] By JP,60-2272,B, in order to obtain isotropic thermal expansion, the cordierite powder and cordierite-ized raw material of an indeterminate form which were compounded beforehand are blended so that it may become a theoretical presentation, and acquiring a cordierite object is indicated by performing shaping and baking. However, this technique was not enough not to make the publication about the porosity of cordierite powder, and particle size, but have collection efficiency important as a filter although it was useful in the thing which has thermal shock resistance thought as important for catalyst support.

[0007] Moreover, ventilating pressure loss is small and it is indicated by by choosing cordierite with a suitable precise grain size marketed as JP,3-68411,A, and using for the aggregate that what reinforcement also has in practical use level can be created. By this approach, in order to make the aggregate precise, cordierite powder with high porosity was re-calcinated, and the process which adds a LAS raw material and carries out eburnation of the cordierite aggregate is indispensable, and there was a problem used as the increase of a large man day.

[0008]

[Problem(s) to be Solved by the Invention] The place which this invention was made against the background of the above present condition, and is made into the purpose is excellent in the ability to regenerate at the time of back wash playback, and is to have a high mechanical strength and offer the compact lightweight nature ceramic filter of cordierite, and its manufacture approach.

[0009]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, the nature ceramic filter of cordierite of this invention (1) With the nature ceramic filter of cordierite for separating solid phase out of a fluid Consist of combination of the nature SERAMMIKU aggregate of cordierite, and a passage septum is formed in a honeycomb configuration. For the both ends, the eye closure is given alternately, the average pore size of a filter is less than [ of the filter aggregate / pitch-diameter  $\times 0.15^{**}5$ micrometer ], the porosity of the whole filter is 30% or more, and the compressive strength of the direction of passage is 100 kgf/cm<sup>2</sup>. It is characterized by being above.

(2) The thickness of a passage septum is 300-2000 micrometers, and it is characterized by the hole density of a filter section cross section being 63 - 71%.

[0010] Moreover, the manufacture approach of the nature ceramic filter of cordierite of this invention (3) The cordierite powder 100 weight section of 30% or more of porosity used as the aggregate, the cordierite-ized raw material 5 used as bridge formation of the aggregate - 30 weight sections, and an ostomy agent are kneaded by adding and mixing a shaping assistant and a solvent below in 30 weight sections, and it is characterized by performing baking after extrusion molding the shape of a honeycomb.

(4) In the above-mentioned manufacturing method, it is characterized by the mean particle diameter of cordierite powder being 20% or less of an extrusion dice slit.

[0011] In the ceramic filter of this invention, hot engine exhaust gas shall be borne by using the nature ceramics of cordierite as the filter aggregate. As for the filter, passage is formed by the honeycomb-like septum. The eye closure of the both ends of a honeycomb is carried out alternately, and the other end of the passage by which the eye closure was carried out is constituted so that opening may be carried out. Exhaust gas passes the septum formed in porosity so that it might enter and mention later from the end of the passage which is carrying out opening of the honeycomb-like filter, reaches the opening edge through adjoining passage, and is discharged out of a filter. When exhaust gas passes a septum, uptake of the dust particle contained in exhaust gas is carried out to a septum. There is an advantage which can take a large filter area per unit volume by forming a septum in the shape of a honeycomb as mentioned above.

[0012] If a ceramic filter is created only from a cordierite-ized raw material, the pore in a filter will turn into bad pore of chain nature like the conventional example shown in drawing 2 . Since pore is formed of the talc in a cordierite-ized raw material, and a silica, this is because it leaves the particle shape of talc and a silica. Playback by the back wash pulse air at the time of back wash playback is not fully made, with the passage of time, pressure loss increases and the bad pore of such chain nature cannot obtain a uniform filter shape.

[0013] In the ceramic filter of this invention, the cordierite particle which should serve as the aggregate was beforehand used as a raw material, and increase of mechanical strength was aimed at by constructing a bridge and connecting this with a cordierite-ized raw material. According to this approach, the pore made between the aggregates formed with cordierite powder becomes what was excellent in chain nature like drawing 1 , and playback by back wash pulse air is easy to be carried out.

[0014] Thus, it sets to the ceramic filter of this invention, and is the porosity as the whole filter the 100 kgf/mm compressive strength of the direction of passage in mechanical strength sufficient also as 30% or more, and a concrete target 2 Maintaining the above, filtration velocity or pressure loss can be made low, and it can have the uptake time amount made useful on industry. On the other hand, if the rise of pore chain nature and porosity is thought as important too much, bridge formation between the aggregates will become less enough, and degradation of mechanical strength will start. It found out this invention person becoming the filter it has a filter and pore chain nature and mechanical strength by making the average pore size of a filter into less than [ aggregate particle diameter  $\times 0.15^{**}5$ micrometer ] various examination and as a result of an experiment, and becoming nature of cordierite ceramic FURUTA of a useful back wash playback type on industry.

[0015] That is, if the pitch diameter of pore exceeds diameter  $\times$  of the aggregate  $0.15+5$ micrometer, a filter septum will be passed to a dust particle, collection efficiency falls, or the mechanical strength of a septum falls. Moreover, the increase of pressure loss, and in order to spoil the chain nature of pore further, as for that in which the pitch diameter of pore is less than diameter  $\times$  of the aggregate  $0.15-5$ micrometer, a back wash property deteriorates. Thickness of a passage septum is set to 300-2000 micrometers in the desirable mode of the ceramic filter of this invention. If the thickness of a passage septum is too small, in shaping of a honeycomb Plastic solid becoming difficult, lack is caused to a mechanical

strength, the uptake capacity of dust becomes small, and it produces un-arranging, such as producing the need of reproducing frequently. Moreover, if the thickness of a passage septum is excessive, pressure loss will increase, and also the fall of the filter area per unit volume (it is called "volumetric efficiency" below) is caused.

[0016] Although the hole density of the filter section influences the volumetric efficiency of a filter, a mechanical strength, pressure loss, etc. in relation to the outer diameter of the filter section, the consistency of passage, passage septum thickness, etc., it makes hole density 63 - 71% in the ceramic filter of this invention. Moreover, if the cordierite powder of 30% or more of porosity is used, by the burning shrinkage at the time of baking, the pore which is inherent in cordierite powder can act as pore between the aggregates, and can obtain the good pore of chain nature.

[0017] The range of the particle diameter of the cordierite powder used as the aggregate is 5-450 micrometers preferably, and it is desirable to consider as 20% or less of value of an extrusion dice slit. By this, a powder particle is fully stuck by pressure at the time of extrusion molding, the moldability of a honeycomb Plastic solid becomes good, and a mechanical strength improves. There is a possibility of a Plastic solid will not only becoming perfect, but fault arising at the time of shaping if the mean particle diameter of cordierite powder exceeds 20% of an extrusion dice slit, and extrusion pressure increasing and producing deformation to an extrusion dice.

[0018] Since cordierite is the ceramics which is hard to sinter, it is not enough as a mechanical strength to have sintered only cordierite powder. In the manufacturing method of the nature ceramic filter of cordierite of this invention, in order to construct a bridge in cordierite powder and to combine a powder comrade, 5-30 weight section addition of the cordierite-ized raw material is carried out as a cross linking agent to the bone agent 100 weight section.

[0019] As a cordierite-ized raw material which should serve as bridge formation of the aggregate, it blends and talc, a kaolin, an alumina, a silica, etc. are used, for example so that it may become a cordierite presentation. When it calcinates only by it, as for this cordierite-ized raw material, it is desirable to use that from which porosity becomes 35% or more. Moreover, it is good, and a thermal-expansion property, thermal stability, and chemical stability act effectively as a cross linking agent by the liquid phase reaction, and this cordierite-ized raw material has them. [ effective in improvement in the free passage nature of pore ] However, if the addition of a cordierite-ized raw material becomes superfluous, the pore which ink jar type blockaded will be formed of it, and the disadvantage crack and back wash ability to regenerate will decline [ a pore comrade's free passage nature ] by it. By adding a cordierite-ized raw material in the predetermined range as a cross linking agent, it excels in a back wash property and the high sintered compact of a mechanical strength is obtained.

[0020] Since an ostomy agent helps to form pore in a sintered compact by oxidizing and evaporating at the time of baking, in addition, the suitable carbon powder of a dimension, polyurethane powder, etc. can be used for it. An ostomy agent can be added in the range below 30 weight sections to the aggregate 100 weight section. A shaping assistant and a solvent can help kneading and extrusion molding of raw material powder, and what is used for extrusion molding of the usual ceramic powder can be used. Although methyl cellulose, diethylene-glycol stearate, etc. are mentioned as a shaping assistant and the class and an addition may be chosen suitably, it is desirable to add more than 3 weight sections in the assistant sum total to the aggregate 100 weight section for the fall of extrusion pressure and the moldability of a Plastic solid.

[0021] The burning temperature of a honeycomb Plastic solid is beyond the temperature to which a cordierite-ized raw material begins to sinter cordierite, and is chosen in the temperature requirement below the melting point of cordierite. If burning temperature is too high, the cordierite aggregate will vitrify and a coefficient of thermal expansion will become large. It calcinates preferably in a 1350-1430-degree C temperature requirement. In addition, the alternate eye closure of a filter end face may be performed before baking of the body of a filter, and may be performed after baking of a body.

[0022]

[Example] Hereafter, an example explains this invention concretely.

(Experiment 1) The cordierite powder which has the presentation shown in Table 1 and physical properties was used as the aggregate, and it prepared at a rate which shows the raw material which has the presentation shown in Table 2 in the column of the blending ratio of coal of Table 2, and considered as the cordierite-ized raw material. Moreover, graphite was used as an ostomy agent. The aggregate, the cordierite-ized raw material, and the ostomy agent were blended as shown in Table 3. In each trial batch, the methyl cellulose 10 weight section and the diethylene-glycol stearate 3 weight section were added as a shaping assistant. Water was added to these as a solvent, mixing and kneading were performed, and it considered as the plastic matter for extrusion molding.

[0023]

[Table 1]

コーライト粉末 種類	成分 (wt%)					平均粒径 ( $\mu\text{m}$ )	気孔率 (%)
	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	NaO		
A	13.7	35.5	50.5	0.1	0.2	65	30
B	13.7	35.5	50.6	0.1	0.1	70	30
C	13.7	35.5	50.5	0.1	0.2	65	25
D	13.7	35.4	50.6	0.1	0.2	66	40
E	13.7	35.4	50.6	0.1	0.2	430	30
F	13.7	35.5	50.5	0.1	0.2	55	30

[0024]

[Table 2]

原料の種類	成分 (wt%)						配合割合 (%)
	lg-loss	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	
タルク	5.7	30.7	1.2	60.9	—	1.0	40.0
カオリン	13.9	—	38.6	45.5	0.8	0.4	25.0
仮焼カオリン	0.1	—	45.1	53.1	0.8	0.4	21.0
アルミナ	0.3	—	99.4	—	—	—	14.0

[0025]

[Table 3]

区分	コーライト粉末 種類	コーライト粉末 (重量%)	コーライト化原料 (重量%)	造孔剤 (重量%)	隔壁厚さ ( $\mu\text{m}$ )	骨材径 ( $\mu\text{m}$ )	気孔率 (%)	細孔径 ( $\mu\text{m}$ )	圧縮強度 (kgf/cm <sup>2</sup> )	圧力損失 (mmHg)	捕集効率 (%)	逆洗特性 (mmHg)
実施例 1	A	95	5	—	301	60	30.2	4.1	115.2	250	98.0	380
実施例 2	A	95	5	30	302	59	40.4	9.5	105.5	215	98.8	340
実施例 3	A	70	30	—	300	60	32.1	7.1	121.8	205	98.0	550
実施例 4	A	70	30	30	302	58	41.9	11.9	108.0	153	91.2	505
実施例 5	D	95	5	—	303	58	37.0	9.2	113.2	210	97.0	360
比較例 1	—	—	100	—	302	—	40.0	15.7	152.0	180	91.0	610
比較例 2	A	100	—	—	300	60	27.2	2.9	98.0	280	98.5	350
比較例 3	A	95	5	40	302	58	44.2	10.2	99.0	204	98.7	320
比較例 4	A	70	30	40	303	59	45.3	12.6	98.0	178	96.0	490
比較例 5	A	60	40	40	301	58	46.0	14.3	103.0	155	80.3	540
比較例 6	B	70	30	—	成形不能		—	—	—	—	—	—
比較例 7	C	70	30	30	298	61	38.9	4.1	116.0	233	96.0	580
比較例 8	C	95	5	30	299	62	32.0	3.8	108.5	252	97.6	390

[0026] They are these plastic matters 1cm the slit width of 330 micrometers 2 Using the extrusion dice of hit 38 cel, extrusion molding was performed to 130mmphix170mm, and it calcinated at 1400 degrees C. After baking, the eye closure was given and the filter sample was obtained. Measurement of septum thickness and the number of cels was performed about the filter sample, and porosity measurement by the decoction method and pore size measurement by the method of mercury penetration were performed. The cylinder sample of 25.4mmphix25.4mm was started from the filter sample, and the compressive strength of the direction of passage was measured. It asked for the diameter of the aggregate by SEM observation.

[0027] Using the measuring device shown in drawing 3, filter shapes are the conditions for pulse period 0.5 seconds, and searched for a back wash property and pressure loss for a part for amount of emission of 3m 3/, back wash pulse \*\* 6 kgf/cm<sup>2</sup>, and pulse-separation 5 minutes. The result is shown in Table 3. Collection efficiency was made into collection efficiency here from test initiation before 3 hours with the ratio of the average uptake time amount of the amount of uptake and yield of a dust particle by which uptake was carried out at the uptake tank 14. A value becomes large rather than the collection efficiency shown here, or 3 hours after shows the same value as these.

[0028] Moreover, pressure loss was measured with the pressure-loss measuring device 12, and pressure loss 3 hours after test initiation was made into the back wash property. What is excellent in a back wash property has a small pressure loss value by time 3 hours, and, as for what has a bad back wash property, this value becomes large. It turns out that porosity exceeds 30% according to Table 3, and, as for the thing within diameter xof the

aggregate 0.15\*\*5micrometer, the pore size of a filter is excellent in compressive strength and a filter shape. Filter pore size of pressure loss is [ the following / diameter x of the aggregate 0.15-5 ] large, and it is clear to become an engine load.

[0029] On the other hand, with the filter which has the average pore size beyond diameter x of the aggregate 0.15+5micrometer, it turns out that collection efficiency falls and it does not act as a useful filter. Moreover, the filter of 30% or less of porosity also has large pressure loss. Moreover, it turns out that it is obtained when these filters carry out 5-30 weight section addition of the cordierite-ized raw material using the cordierite powder of 30% or more of porosity and an ostomy agent is made below into 30 weight sections. When a cordierite-ized raw material is made below into 5 weight sections, the fall of compressive strength not only takes place, but bridge formation between the aggregates becomes insufficient and increase of pressure loss takes place. Moreover, in the addition more than 30 weight sections, enough, the bridge formation between the aggregates becomes what has the gestalt of pore close to bad drawing 2 of chain nature, although a next door and compressive strength rise, and it turns out that back wash nature deteriorates. Moreover, if an ostomy agent is added more than 30 weight sections, although porosity will rise, it becomes what bars bridge formation between the aggregates, and it turns out that compressive strength falls.

[0030] (Experiment 2) Various extrusion slit width and cel configurations were changed, and various ceramic filters were manufactured by the same process as experiment 1 except it. Cordierite powder, a cordierite-ized raw material, an ostomy agent addition, and each measurement result are shown in Table 4.

[0031]

[Table 4]

区 分	コルディエライト粉末 種 類	コルディエライト粉末 (重量%)	コルディエライト化合物 (重量%)	造孔剤 (重量%)	隔壁厚さ ( $\mu\text{m}$ )	骨材径 ( $\mu\text{m}$ )	セル数 (1/ $\text{cm}^2$ )	開孔率 (%)	気孔率 (%)	細孔径 ( $\mu\text{m}$ )	圧縮強度 ( $\text{kgf}/\text{cm}^2$ )	圧力損失 ( $\text{cmH}_2\text{O}$ )
実施例 2	A	95	5	30	302	59	31.0	70.8	40.4	9.5	105.5	215
実施例 6	A	95	5	30	301	59	43.5	63.3	40.5	9.6	128.4	252
実施例 7	A	95	5	30	2003	59	0.6	71.0	40.3	9.5	105.3	255
実施例 8	A	95	5	30	434	59	15.1	68.9	40.5	9.4	113.6	220
比較例 9	A	95	5	30	300	60	15.5	77.8	40.3	9.4	78.1	180
比較例 10	A	95	5	30	300	60	62.0	53.3	40.4	9.4	144.7	268
比較例 11	A	95	5	30	2098	59	0.6	69.7	40.4	9.4	103.5	264
比較例 12	E	95	5	30	2001	398	0.6	70.8	43.2	60.4	100.3	240
比較例 13	F	95	5	30	250	49	62.0	64.5	37.3	7.8	98.0	240

[0032] That to which septum thickness exceeds 2000 micrometers has pressure loss higher than Table 4, and it turns out that the thing 300 micrometers or less does not have the reinforcement of the level which compressive strength falls and can be carried in an automobile. Moreover, even if it is in the range of these septa thickness, pressure loss is large and hole density of a filter cross section cannot real-use it, although the compressive strength (with [ it excels in collection efficiency and ] no :data) of 63% or less of thing is enough. If a numerical aperture exceeds 71%, since compressive strength will fall and filter effective area will also become small further, it turns out that it cannot become a useful filter on industry.

[0033]

[Effect of the Invention] By the nature ceramic filter of cordierite and its manufacture approach of this invention, collection efficiency is high, pressure loss is small, it is lightweight and compact, and the nature ceramic filter of cordierite excellent in the back wash property which has the reinforcement which can be carried in an automobile, and its manufacture approach can be offered.

[Translation done.]

